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By George N. Pulley

Bureau of Chemistry and Soils, U. S. Citrus Products Station, Winter Haven, Fla.

The possibility of utilizing grapefruit residue for the production of naringin and its hydrolytic products, together with the fact that there is a limited commercial production of naringin at the present time, has shown the desirability of determining the solubility of naringin in water at various temperatures.

Naringin (C27H32O142H2O) was discovered by DeVry (2) in the flowers of grapefruit trees growing in Java. Will (6,7,8), Zoller (9), and Asahina and Inubuse (1) have conducted studies to determine its properties. Its increased solubility in hot water or juice has been noted by Fellers (3), and Segal and de Kiewiet (5) in technological studies on grapefruit products. The content of naringin in both peel and juice appears to diminish as the fruit matures. It is soluble in alcohol, acetone, and water. When crystallized from these solvents and dried at 110°C. it melts at 171°C. When crystallized from water it has an additional 6 molecules of water and melts at 83°C. The bitter taste of naringin is pronounced: a water solution containing one part in ten thousand has a distinctly bitter taste.

The naringin used in these experiments was made from grapefruit peel, purified by the method outlined by Poore (4) and dried at 110°C.; the melting point was 171°C. (uncor.).

The solubility of naringin was determined by adding an excess of the purified material to 150 cc. of distilled water contained in a flask which was closed with a rubber stopper and immersed in a constant-temperature water bath. The flask was left in the bath 2 hours and was shaken every 15 minutes. At the end of 2 hours the solution in the flask was rapidly filtered, using a water-jacketed funnel. A measured volume of the clear filtrate was transferred to a weighed evaporating dish and evaporated to dryness over a steam bath, then dried at 110°C., cooled, and weighed. The amount of naringin dissolved per 1000 cc. was calculated from the average of two determinations.

The solubility at 6°C. was determined by placing the flasks in an electric refrigerator, while the solubility at 20°C. was determined in an ice-cooled box. The variation in the temperature at these two points was greater than at the higher temperatures, but, since the increase in solubility of naringin between 6° and 35° is so small, fluctuations in temperatures at 6° and 20°C. would have no significant effect upon the solubility value. Solubilities at other temperatures were carried out in a water bath, the temperature of which was controlled by means of a gas thermoregulator. The water in the bath was kept in constant motion by means of compressed air.

The data given in Table I and Figure I show that up to 45°C. the increase in solubility with increase in temperature is not pronounced. From 45°C. to the melting point (83°C.) the solubility increases rapidly with increase in temperature.

(1) Asahina, Y., and Inubuse, M., J. Pharm. Soc. Japan, 49, 1928-35 (1929).

(2) DeVry, Jahresber, Pharmacog. 1886, 132.

(3) Fellers, C. R., Canner, 69, No. 18, 11-12 (1929).

(4) Poorc, H. D. Ind. Eng. Chem. 26,637-9 (1934).

(5) Segal, B., and de Kiewiet, T., J. South African Chem. Inst. 14, 43 (1931).

(6) Will, W., Bor., 18, 1311-25 (1885).

- (7) Ibid., 20,294-304 (1887). (8) Ibid., 20, 1186 (1887).
- (9) Zoller, H. F., Ind. Eng. Chem., 17, 1065 (1925).

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TABLE I. SOLUBILITY OF NARINGIN IN WATER

Temperature	of	Water		Solubility	in	Water
° C.				G./10	000	cc.
6				0.1	17	
20				0.:	50	
35		0.79				
45	1.96					
55	7.16					
65		42.21				
75		108.24				

The decreased solubility of naringin at low temperatures may at times cause the precipitation of this substance in canned grapefruit juice and sections, as has been pointed out by Fellers and by Segal and de Kiewiet (5). This is especially true if the juice or sections have been prepared from immature or frozen fruit. In the case of canned juice the glucoside generally settles to the bottom of the container as a yellow sludge. Sections may show light yellow spots, which macroscopically have the appearance of mold. At times the juice has a milky appearance due to minute crystals of naringin.

